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TEST METHODS FOR INJECTABLE BARRIERS

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16. ABSTRACT

Grouts are becoming increasingly important in producing barriers to contaminated ground-water flow at hazardous waste sites. Grouted barriers can be used at depths and under conditions where slurry trenches are impractical. To employ grouts to advantage at waste sites it is necessary to select materials that solidify or gel in the presence of industrial waste compounds that may be present; that are unaffected by aggressive chemical wastes; and that create impermeable barriers when injected into permeable units.

A wide variety of test procedures have been employed in bench and pilot-scale evaluation of grouts. Many testing systems do not duplicate field conditions closely enough to be useful. A standard approach using gelling time determinations, static chemical durability testing and permeability determinations on cores of grouted material is suggested.

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TEST METHODS FOR INJECTABLE BARRIERS

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ABSTRACT: Grouts are becoming increasingly important in producing barriers to contaminated ground-water flow at hazardous waste sites. Grouted barriers can be used at depths and under conditions where slurry trenches are impractical. To employ grouts to advantage at waste sites it is necessary to select materials that solidify or gel in the presence of industrial waste compounds that may be present; that are unaffected by aggressive chemical wastes; and that create impermeable barriers when injected into permeable units.

A wide variety of test procedures have been employed in bench and pilot-scale evaluation of grouts. Many testing systems do not duplicate field conditions closely enough to be useful. A standard approach using gelling time determinations, static chemical durability testing and permeability determinations on cores of grouted material is suggested.

KEY WORDS: Grout, gelling time, compatability, permeability

Sealing materials pumped or injected into rock and soil have received widespread attention in the construction industry for control of ground-water and surface water infiltration. Grouting with chemical or particulate grout

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to control water movement has been routine procedure in remedial actions undertaken at dams or in tunnels [1, 2, 3]. Applications of grouts in waste control are becoming more common at sites where the depth to an impermeable horizon or presence of coarse (boulder-sized) particulate material makes slurry trenching impractical. Large-scale vertical grout curtains have been employed in several waste control projects [4, 5] and have been planned or evaluated at several others [6, 7].

As grouts are more commonly employed in hazardous waste containment, it is necessary to develop techniques for testing grouting material used at hazardous waste sites to determine:

(a) the ability of the grout to successfully gel or set under adverse chemical conditions.

(b) the ability of the gel to remain intact when exposed to aggressive chemical compounds.

(c) the reduction in permeability that can be produced in grouting reactions.

The purpose of this paper is to examine techniques that have been employed in testing grouts used for hazardous waste containment and to suggest how testing methods can be altered to better evaluate materials and methods used in producing injected barriers. This review concentrates on procedures used in laboratory evaluation of materials and small-scale field tests.

A summary of the major grout types that are available and their properties are given in Table 1. The materials vary from clay slurries that thicken or gel as water is lost to surrounding materials to low-viscosity organic materials that go through a complex series of linking reactions to form organic polymers. In all cases, the grouts become effective by forming a non-leaching solid or semi-solid that blocks the pore spaces in the permeable medium (for example,

TABLE 1 - Major grout types with potential application in waste containment

Type of Grout*	Major Components	Chemical Compatibility
Portland Cement/Clay (Particulate)	Bentonite and/or Portland Cement	Resistant to most waste except strong acids or organics.
Sodium Silicate	Sodium silicate and organic acids or esters	Resistant to most acids and organics, attacked by bases.
Acrylates	Acrylate monomer cross-linking agent and initiator and accelerator	Resistant to most organics, attacked by acids, bases and salts.
Furan	Furfuryl alcohol, filler and acid	Resistant to all but oxidizers.
Lignosulfites	Calcium lignosulfonate, acid and sodium dichromate	Attacked by strong reducing agents
Urethanes	Urethane monomer and water	Attacked by aldehydes, ketones, acid and bases.
Phenoplasts	Resorcinal, formaldehyde, and sodium hydroxide	Attacked by hydrocarbons and bases.
Aminoplasts	Urea and formaldehyde	Attacked by acids, bases and ethers or epoxides.

* Data from References 8, 9

a sand or silt) in which it is injected. The successful use of grout may directly depend on the ability of a grout to penetrate the matrix material before gelling. In the case of particulate grouts such as cement or bentonite (clay) the pore size of the grouted unit must be relatively large (above that found in silt) in order for the grout grains to move between matrix particles. Where solution grouts (usually referred to as chemical grouts) are used the grout with the lowest viscosity has the best ability to penetrate. Because most groutable materials below a waste site contain water, viscosities near that of water are desirable. The grouts that have the widest application in grouting both coarse and fine-grained sediments are chemical grouts. Portland cement and clay grout are limited to coarse sand or gravel unless a cavity is produced by jetting or hydrofracting (splitting the substrate with pressurized grout) techniques.

Chemical Compatibility during Grout Setting

Most of the materials employed in grouts are organic polymers, such as acrylate or urethane, or soluble silicates, such as acidic or basic sodium silicates (Table 1). The gelling or polymerizing reactions in the various grouts can be slowed or stopped by a variety of chemical compounds found in industrial wastes. Many gelling reactions can also be accelerated by chemical compounds occurring in the permeable materials being grouted. These accelerated gelling or flash setting reactions can make it impossible to distribute grout in a permeable unit before hardening occurs. Table 2 summarizes some of the more common interfering reactions occurring in grouts. Not all of these reactions would occur in grouting chemically contaminated soil or rock units; but the high probability of some interaction of waste and grout make laboratory testing for abnormal gelling reactions necessary.

Laboratory gel time determinations are approximate tests based on the

Table 2 - Common Interfering Reactions that Occur in Solidifying Grouts

Type of Grout	Interfering Reaction
Portland Cement/Clay (Particulate)	Organics can attack clays, acids destroy lime in portland cement
Sodium Silicate	Acids and inorganics can cause flash sets, alkali can prevent gellation
Acrylates	Strong oxidizers and reducers can interfere with gellation by destroying initiators
Furan	Strong reducers can stop gellation
Lignosulfites	Strong reducers can destroy the gelling initiator
Urethanes	Ammonia compounds compete for the prepolymer and form urea compounds
Phenoplasts	Strong acids can stop gellation
Aminoplasts	Alkalis prevent gelling

time required for the grout to become too viscous to pour or too stiff to stir easily. Mechanical testing equipment to detect changes in viscosity is available; but has limitations. Available equipment includes paddle gelometers [10, 11] rotational viscometers [12] or penetrometer set measuring devices [10]. None of these measuring devices have proved completely satisfactory because of the gradual stiffening of chemical grouts and the lack of resistance to penetration observed in many chemical gels. Problems also arise from the shearing of the newly-formed gel that can reliquify part of the solid and give a false reading of the viscosity.

The simplest techniques for judging laboratory gel time for grouts are the "glass rod method" and the "tilting tube method." The "glass rod method" involves drawing a glass rod across the surface of the liquid material and noting the time elapsed when the mark produced by the rod will remain on the surface for several seconds. The "tilt tube method" involves observing the time elapsed from grout mixing to the point at which the grout will no longer pour from a test tube [12]. Neither method is automated and constant attention is needed to obtain accurate results.

None of the commonly-used gel time indicators accurately mimics the behavior of the grout in the field. Large-scale laboratory tests have shown that silicate grouts, for example, can be pumped into dry sand test beds for five to six times longer than the laboratory-determined gel times before viscosity increases become evident. The shearing of the grout in the pumps and hoses and in the sand test bed probably keeps the grout fluid longer than the typical gel time for stationary material [13].

Laboratory gel times are a valid technique for detecting problems related to unusual set times due to background chemical interaction between pore fluids and grout, but the use of laboratory set times in field operations may

result in consistently underestimating gel times.

Chemical wastes can be introduced into grout in a variety of ways. In field operations in sealing a waste site, the grout would be injected and would displace and partly mix with chemically-contaminated water. A simple laboratory approach aimed at reproducing the field situation is to mix equal parts of the grout and the waste or simulated waste uniformly and to observe any changes in gelling time. This mixing procedure does not exactly duplicate field conditions where the advancing grout would not be well mixed with the chemical waste it encounters, but the test does provide a baseline condition for estimating interferences. The test program involving direct mixing of equal volume of grout and waste has been effective in demonstrating that a variety of compounds potentially present in industrial waste can affect grout gel times even when present in pore water at concentrations below 10% by volume. Effects observed vary from a flash set of silicate grout with 10% copper sulfate solution to complete retardation of any gelling (10% ammonium hydroxide solution with urethane grout) [10]. Testing of retardents in portland cement is generally done in a similar way, by introducing the chemical of interest directly into the cement mix.

The concentration of contaminants in the grout can be varied to observe how the change in gel times is related to chemical interference during the setting reaction. Many waste materials will prevent gelling in grouts by destroying compounds added as initiators or accelerators. Other waste compounds can initiate competing reactions in grout that proceed faster than polymerization, so that reactive monomer is used up without any solid (or with a very weak, poorly-polymerized solid) forming.

No standard methods exist for testing chemical interferences in grouts, but the general approach has been to determine gel times for grouts with the likely contaminants being mixed directly with the grout. More thorough approaches

involving the systematic measurement of reduced strength or reduced permeability of grouted sand samples could also be used to produce realistic and applicable data.

Chemical Compatibility after Barrier Formation

Most grout barriers are put in place to last indefinitely. However, grouts are subject to chemical attack just as flexible membrane liners or compacted clay liners would be. The question of chemical stability of grout materials is a serious one. Components in industrial waste can cause grout barriers to shrink, swell, dissolve or become brittle. Grouts layers can be either leached away [14] or be added to when ground water and wastes reacts with the grouted unit.

Two approaches have been commonly employed to test hardened solidified polymer durability in a reactive chemical environment. One approach has been to expose specimens of grout to wastes or simulated waste in a static test and to observe any changes; such as shrinking, swelling, cracking or discoloration that might indicate the grout polymer is breaking down. This type of static exposure test has been used to test grouts but no standard test conditions are used.

The results of a 16-week test on acrylate gels exposed to 10% solutions of alcohols, ketones, hydrocarbons acids and metal salts has been reported by Clarke [15]. A 20-day test on silicates, urethanes, acrylates and portland cement with acids, bases, hydrocarbons and metal salts has been reported by Larson and May [10]. In the latter test 10% (by volume) solutions or saturated solutions (for materials with water solubilities below 10%) were employed.

A one- to nine-month durability test undertaken on sodium silicate-grouted sand specimens was reported by Lord et al. [16]. The simulated wastes used were generally highly concentrated or saturated solutions (for example, 100% by volume sodium hydroxide and saturated sodium dichromate). The time

for disintegration was arbitrarily taken as the time needed to observe an easily noticeable number of sand grains on the bottom of the test vessel.

Many important parameters such as the volume- to-surface area ratio for the test specimens, presence or absence of supporting material (soil or rock) and the types of supporting material added change from one reported procedure to another. The volumes and concentrations of waste or waste simulants used and the frequency with which the solutions are renewed are not standardized. At the present time no uniform protocol that can be used for static durability testing of grouts exists.

A second approach to testing grout durability involves the use of flow-through tests, where the test specimen of grouted material is placed in a permeameter and water containing the potentially deleterious waste is forced through the sample. The effect of the permeant is observed by noting changes in the permeability of the test specimens (although permeability changes may not always reflect grout deterioration).

Several significant problems are involved in using a flow-through test system. If a sample is prepared in such a way that the particulate material is saturated (or impregnated) with a grout and all the void space is filled; the permeability will be extremely low and a true flow-through test becomes nearly impossible. For example, Doiron, Burkhard and White [17] prepared samples of sand grouted with acrylamide that had permeabilities of 0.2×10^{-9} cm/sec. The sand was saturated with acrylamide and vibrated before gelling took place to consolidate the sand. Flow-through testing would be ineffective with such highly impermeable materials because only one surface would be effectively exposed to the permeant in any reasonable test period.

Other problems arise if the chemical permeant being used reacts with the grout to produce a precipitate or gas bubbles. The precipitate can clog the permeameter and prevent measurements from being made [16]. Gas bubbles produce

an unsaturated condition and cause inconsistent, erratic permeability measurements.

When flow-through chemical compatibility tests are used, the samples are typically saturated with water or a dilute calcium sulfate solution. After a reference hydraulic conductivity is established; the chemical permeant is introduced into the sample [16, 18]. However, as the permeant changes the measured hydraulic conductivity will change because the kinematic viscosity of the new permeant is different from the baseline liquid (water or a dilute calcium sulfate solution). The higher the kinematic viscosity of the permeant the lower the permeability measured. To understand any permeability changes caused by chemical interaction between the grout and the permeant, it is necessary that any changes in the kinematic viscosity of the permeant be accurately determined.

The complications involved in flow-through compatibility tests make flow-through methods complex to use and limit the technique to the examination of materials (grouts and permeants) that do not react to produce gas or precipitate. Additionally, any changes in the viscosity of the permeant caused by dilution with interstitial water or interaction with grout must be determined independently so that the changes in the permeability of the grout sample can be documented. Static testing systems are less complex, but require consideration of a large number of variables related to sample geometry and the characteristics of test solutions. Standard test protocols for both static and flow-through tests are needed if useful data are to be developed.

Permeability Testing

Permeability tests on grouted material can produce widely different results when different sample preparation techniques are employed. In a field grouting situation, grout would be pumped out from a point or line source and would form a sphere or cylinder of grouted media [13]. Grout cylinders or

grout bulbs (Fig. 1) may have irregular shapes that reflect slight variations in the pump pressure, media permeability and grout viscosity. In many laboratory preparations, efforts are made to produce the most complete grout penetration possible, regardless of the limitations of field application. For example, in Lord et al. [16] silicate grout was thoroughly mixed with Ottawa sand and then compacted into molds with a Harvard compactor. Dorion, Burkhard and White [17] (as previously mentioned) mixed a well-graded, washed, medium sand with acrylamide and mechanically vibrated the sample to assure maximum density was achieved. Spalding, Hyder and Munro [7] prepared test samples by passing three pore volumes of grout through a packed soil test column; then the column was plugged before gelling of the grout occurred so that no grout could drain out. These techniques assured that all voids were filled. Samples prepared in the three studies referenced had extremely low permeabilities (usually at the limit of measurement of the test equipment); but these tests results bear little relation to what will occur in the field.

Vinson [19] and Vinson and Mitchell [20] developed a preparation technique for grouted sand samples that closely approximates the behavior of grout in the field. The grout, a polyurethane in this report, was injected into a sand-filled mold and a sphere or cylinder of grouted material was produced. Both dry and saturated sands were used. The grouted sand cylinders were used for permeability testing. The limited space available for the expansion of the grout in the cylindrical molds did result in the specimens prepared as cylinders having slightly higher densities than the samples prepared as spheres; but the densities overlapped enough to indicate that the grout distribution in the cylinders was approximately what would be obtained if the grout had been allowed to flow outward with no cylinder walls present.

The bench-scale work was verified in a series of larger-scale tests where

each test run involved injecting 9 kilograms of grout into 0.2 cubic meters of sand packed in a steel drum. A similar test system is shown in Figure 2. Cores, 3.5-cm in diameter, were cut from the large grout bulbs produced in the tests. The permeabilities of the cores were comparable to those obtained from the injected cylinders. Of all the sample preparation systems employed to produce grouted sand test specimens, the cores from large grout bulbs represent the samples that most closely approach field conditions.

The laboratory procedures for determining permeability of grouted materials have generally followed standard triaxial permeability test protocols, where the sample is fully saturated and back pressure is employed as recommended by Zimmie [21]. Where fixed wall permeameters have been employed [7, 16], the samples were grouted directly in the test cell.

Field tests to determine permeability of grouted waste using monitoring wells established in the grout-modified soil horizon have been reported by Spalding, Hyder and Munro [7]. In this investigation, the wells were in place prior to grout injection and grout flowed through the screens into the wells. Slug tests on the wells showed that many of them were completely sealed especially where the trench had been dry when the grout was injected. This procedure documented what happened to the wells, but did not directly measure the effect in the grouted wastes.

Alternative procedures that would provide more useful field verification would include coring and recovering a section of grouted material for permeability testing or drilling and setting casing in the grouted horizon for in situ permeability tests [22]. Where conditions warrant, both types of tests could be performed.

Recommendations

Grouts are receiving wide attention in planning remedial action at

hazardous waste sites because they can be used at greater depths and in geologic settings where slurry walls are not practical. In order to use grouts in waste containment it is necessary that standard approaches be developed for testing grouts to select materials that:

a. set or gel in the contaminated ground water often encountered at a waste site.

b. do not disintegrate or dissolve under the chemical conditions at a waste site.

c. seal the treated layer sufficiently to assist in controlling ground-water flow.

The test approach that best answers the question of setting and variation in setting time caused by interaction with waste is the "tilt tube method" used with ground water from a specific waste site or suitable waste simulants. The major disadvantage of using the "tilt tube method" is the difficulty in judging the gel point. Most results from gelling time studies are intended to be used for comparison of one grout mixture to another. In this type of comparative study, approximate results are adequate.

Durability studies on grouts to be used at hazardous waste sites can best be conducted in static tests, where the samples of grouted materials are soaked in contaminated ground water or solutions prepared to simulate contaminated ground water. Flow-through durability tests are difficult to conduct and interpret due to the low permeability of many laboratory samples and the production of precipitates or gases that change the apparent permeability of the samples.

Determinations of the permeability of grouted material can be conducted on specimens prepared using a variety of techniques. The most realistic permeability determinations are tests performed on cores prepared from large

pods of grout injected into dry or saturated sand. The distribution of grout in pods or bulbs probably comes closest to the distribution occurring during field injection. Standard triaxial, upflow, permeameter tests appear to provide dependable data for grouted materials. Field permeability measurements should be performed using an accepted slug or pump test with casing set to the grouted horizon.

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Figure 1. Grout bulb produced by injecting sodium silicate into a simulated water/waste mixture.

Figure 2. Top of grout test bulb produced by injecting sodium silicate grout into a 200-liter drum filled with sand saturated with a simulated water/waste mixture.

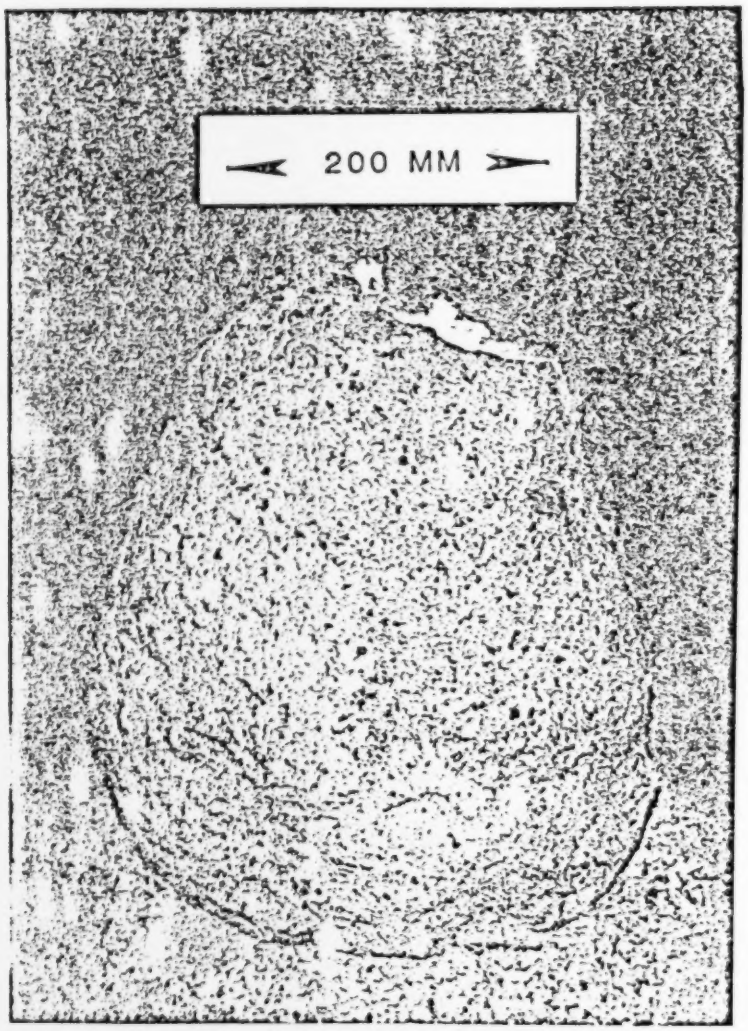

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Figure 1. Grout bulb produced by injecting sodium silicate into a simulated water/waste mixture.

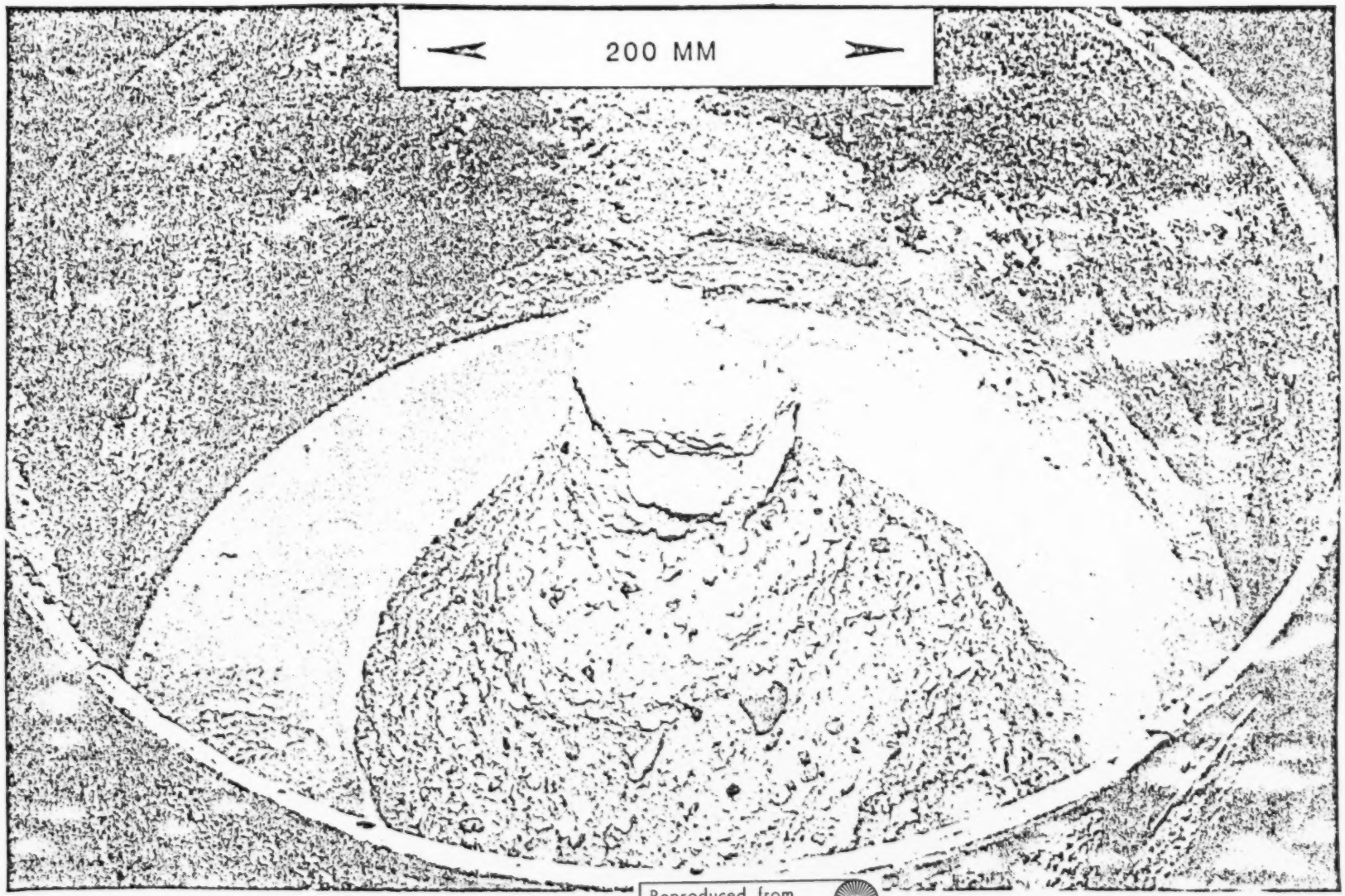

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Figure 2. Top of grout test bulb produced by injecting sodium silicate grout into a 200-liter drum filled with sand saturated with a simulated water/waste mixture.